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Gradient nanostructured surface of a Cu plate processed by incremental frictional sliding

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Abstract. The flat surface of a Cu plate was processed by incremental frictional sliding at liquid nitrogen temperature. The surface treatment results in a hardened gradient surface layer as thick as 1 mm in the Cu plate, which contains a nanostructured layer on the top with a boundary spacing of the order of 100 nm. The boundary spacing increases with increasing distance from the surface, and is accompanied with a decrease in hardness from 2179 ± 31 MPa in the topmost surface layer to 568 ± 10 MPa in the undeformed matrix.

1. Introduction

Surface modification of metallic materials by introducing a gradient nanostructured surface layer is an effective approach to enhance the strength and service lifetime of materials, as demonstrated in many investigations over the past decades [1]. Various techniques have been developed to produce such surfaces [2–6], but there is room for technique development in terms of flexibility in sample geometry, thickness of the hardened layer, efficiency of the processing, etc. This development must be followed up and underpinned by quantitative characterization and analysis of microstructures and properties, as is also part of the present study. In the present work, a technique called incremental frictional sliding is developed and applied to process a Cu plate, which results in thick surface layers with a gradient nanostructure.

2. Experimental

The experimental setup of the incremental frictional sliding has a pin-on-disk configuration as schematically illustrated in Figure 1a. A tool with a round hard tip is pressed against a Cu plate. While the Cu plate rotates, the tool repeats a back-and-forth movement in the radial direction of the plate. As a result, progressive deformation of the entire surface of the plate is achieved. The tool is constructed of a bearing ball of 3 mm in diameter, a steel tube which contains the ball, and a supporting steel rod inside the tube which presses the ball against the end of the tube where the inner diameter of the tube is made smaller than the diameter of the ball (Figure 1c). During the treatment only the ball on the tip of the tool contacts the plate. In the present configuration, the ball is fixed between the steel tube and the supporting rod without any relative rotation, and therefore it rubs the surface of the plate during the processing. For comparison, the tools in conventional deep rolling processes use a similar configuration, but in that case the ball is supported by high pressure fluid in the tube which allows the ball to rotate and roll on the surface of the plate during processing.



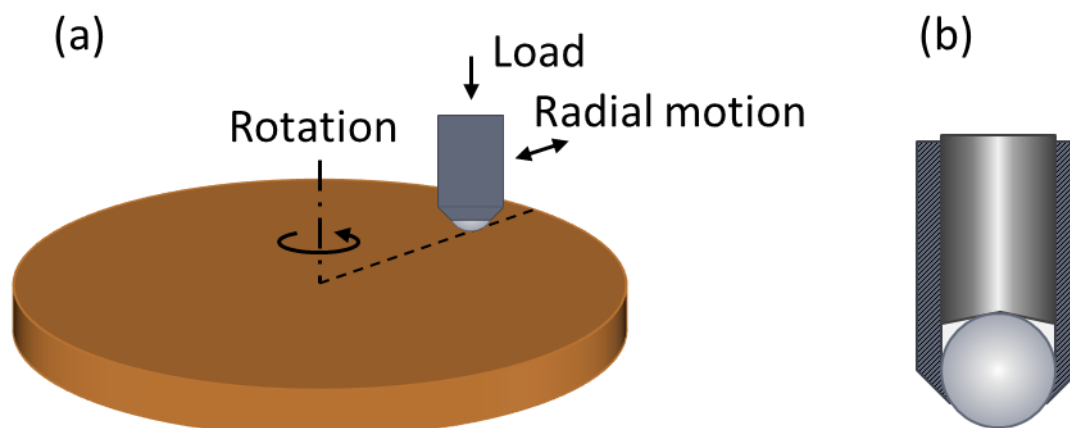


Figure 1. (a) A schematic illustration of the experimental setup for incremental frictional sliding of a Cu plate. (b) A schematic illustration the construction of the tool in (a). The containing tube is cut in cross-section to reveal the bearing ball and the support rod inside the tube.

The plate, 100 mm in diameter and 5 mm in thickness, is made of oxygen free high conductivity Cu with a purity of 99.95%. The plate was annealed at 600 °C for 2 hours prior to the surface processing, resulting in a fully recrystallized structure of tens of micrometers in grain size. During the processing the rotation speed of the plate is 200 rounds per second. To maintain low temperature, the processing is done in 25 interrupted sessions. For each session, the plate was first submerged in liquid nitrogen until the equilibrium temperature was reached, and then with its top surface exposed to the air, the plate is processed for a relatively short duration of 2 minutes during which the tool moved back and forth in the radial direction for about 100 times while the plate was rotating.

Vickers hardness tests and microstructural observations of the processed plate were carried out on the cross section perpendicular to the radius of the plate, at locations about 25 mm from the center of the plate (Figure 2a). Prior to preparation of metallurgical samples, the processed surface was protected by electroplating of Cu, which was done at 32–35 °C for 40 hours and using a solution composed of $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ (72.7 g/L), H_2SO_4 (225 g/L) and NaCl (98.7 mg/L). Metallurgical samples were prepared by mechanical polishing, with a colloidal silica suspension of 0.04 μm in particle size used in the final polishing step. Hardness tests were carried out on a Struers DuraScan-70, using a load of 0.245 N (25 gf). To represent the hardness at a given depth, 2–3 indents were measured. The smallest indent obtained in the present work has a diagonal length of 14.3 μm in the topmost layer, and the largest indent has one of 29.1 μm in the undeformed interior. Due to the limitation of minimum indent size, the smallest depth where hardness was possible to be measured is 8 μm from the surface. The microstructure was investigated using both electron channeling contrast (ECC) and electron backscattered diffraction (EBSD) on a Zeiss Supra 35 scanning electron microscope. A step size of 20 nm was used for EBSD at depths less than 59 μm , where the modal boundary spacing are less than 200 nm, and a step size of 50 nm was used at larger depths corresponding to larger modal boundary spacing.

3. Results

Processing of the Cu plate using the present technique results in substantial microstructural refinement associated with significant hardening below the surface of the plate. Figure 2b shows a typical ECC observation of the microstructure at a low magnification. Within about 100 μm below the surface, the microstructure is too fine to be resolved by ECC at this magnification so that it appears as a dark gray layer without detailed microstructural features. From about 100 μm to 1mm from the surface, the microstructure evolves from an obviously deformed grain structure to a barely deformed coarse-grained structure. Figure 2c shows the hardness in the plate as a function of depth from the surface.

The plate shows a steep hardness gradient down to a depth of 1 mm from the surface. The hardness has the highest value of 2179 ± 31 MPa in the layer just below the surface, and decreases with increasing depth until it reaches the initial hardness of the undeformed material, 568 ± 10 MPa, at a depth of about 1 mm.

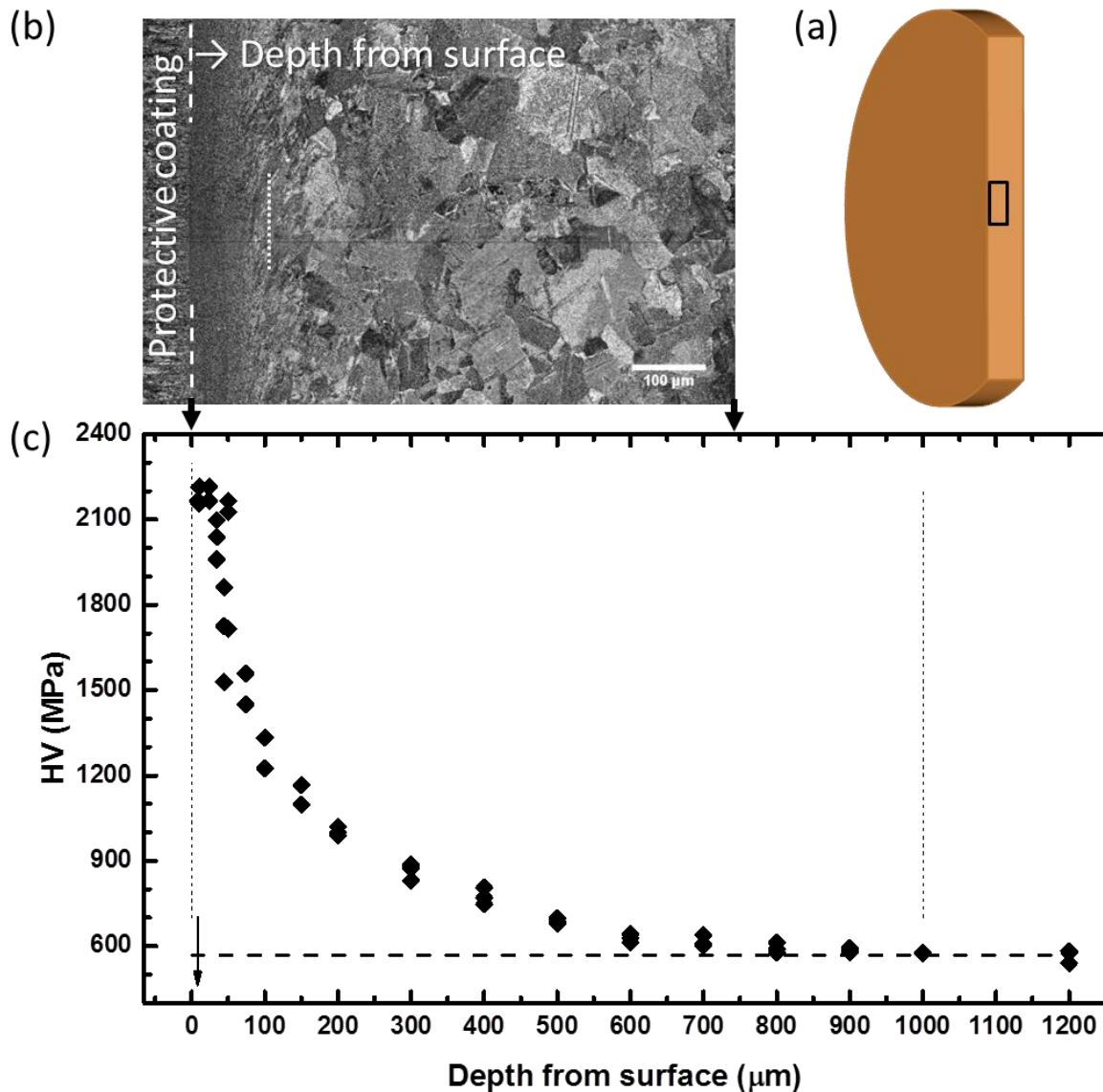


Figure 2. (a) A schematic illustration of the cross-section for the microstructural observations and hardness tests. The rectangle indicates the location of observations in the present work. (b) An ECC image showing the microstructure in the processed Cu plate. The vertical dashed lines mark the surface of the plate. The dotted line roughly marks the thickness range of the dark gray layer. (c) Vickers hardness as a function of depth from the surface. The horizontal dashed line indicates the initial hardness of 568 ± 10 MPa. A pair of vertical dashed lines indicates the thickness range of the hardened layer. The arrow marks the smallest depth (8 μm) where hardness was measured. Note the correspondence between the image in (b) and (c) as marked by a pair of arrows.

The detailed microstructure in the dark gray layer in Figure 2b was further investigated by EBSD with high spatial resolution. In the topmost layer a nanogained structure is developed, as shown in the

EBSD map in Figure 3a. Most of the grains are slightly elongated along the direction of the plastic flow, which is parallel to the surface, and have an aspect ratio of 2.0–2.6. For measurement of boundary spacings, grains in EBSD maps were detected using a critical misorientation of 2° while grain boundary completion using a minimum misorientation of 1° was allowed. Figures 3b and c present statistics of the boundary spacing of the grains measured from Figure 3a. The statistics in Figure 3b use a probability represented by the number of grains, and the distribution shows a modal boundary spacing of 27 nm. The statistics in Figure 3c use a probability represented by the area fraction of grains (which is consistent with the volume fraction of the grains according to the Delesse principle), and the modal boundary spacing is 94 nm. Both statistical methods have been used for measurement of grain sizes of nanostructured materials. For a given microstructure, the modal boundary spacing determined using a probability based on the number of grains is necessarily smaller than the spacing determined based on the area fraction of grains, since larger grains consume a larger area than smaller grains given the same number of grains. However, for a material composed of grains with variation in sizes (and thus with variation in mechanical properties), since the overall mechanical properties are related to the properties of the composing grains via their volume fractions, by e.g. using the rule of mixtures, it is more reasonable to use the latter way of statistics when the mechanical property is of concern. Note that the smallest grains that can be detected by EBSD is limited by the step size (20 nm), and therefore the probability of grains with a boundary spacing smaller than 20 nm are expected to be smaller than the actual value. Nevertheless, both statistical methods show a modal boundary spacing less than 100 nm and indicate the existence of a well-defined nanostructure.

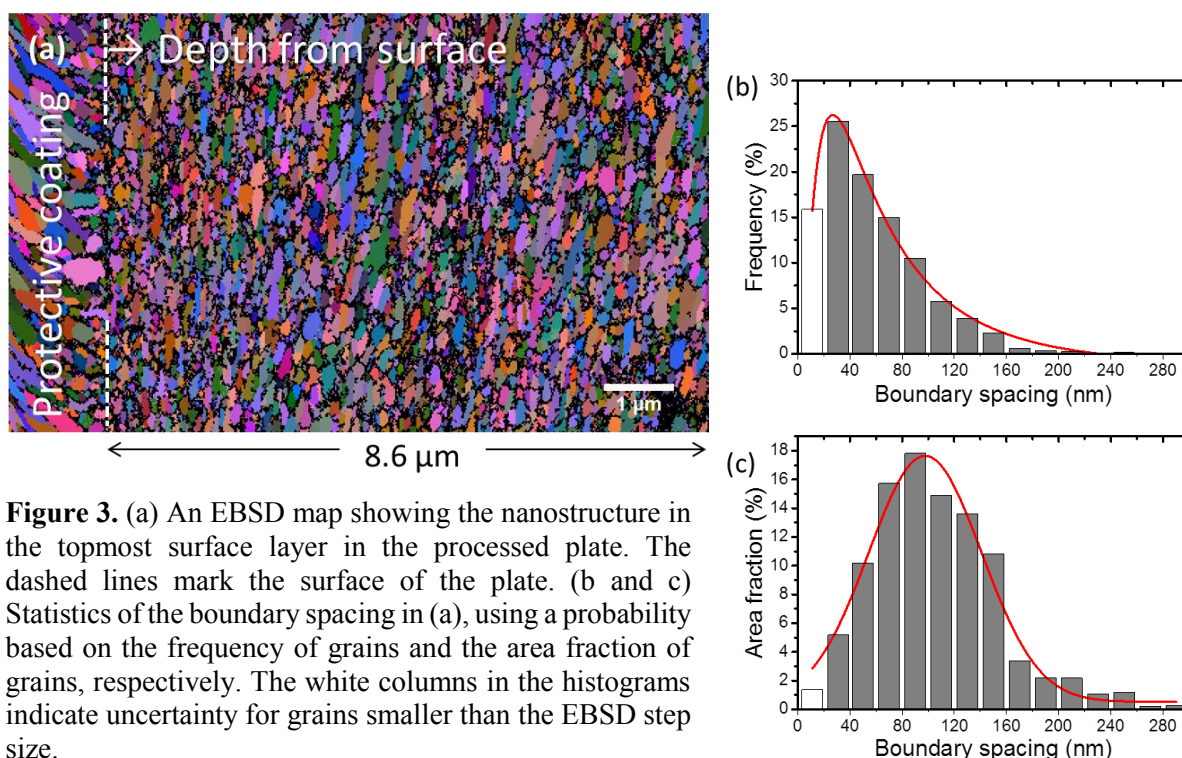


Figure 3. (a) An EBSD map showing the nanostructure in the topmost surface layer in the processed plate. The dashed lines mark the surface of the plate. (b and c) Statistics of the boundary spacing in (a), using a probability based on the frequency of grains and the area fraction of grains, respectively. The white columns in the histograms indicate uncertainty for grains smaller than the EBSD step size.

Figure 4a shows a closer ECC investigation of the dark gray layer in Figure 2b. To explore the variation of boundary spacings as a function of depth, EBSD maps at various depths were obtained. EBSD maps of three typical regions marked in Figure 4a, are shown in Figures 4c, d and e, which covers depth ranges of 0–8.6 μm, 27.6–56.3 μm and 59.0–74.5 μm, respectively. Based on the EBSD maps, the boundary spacing of grains as a function of depth were measured from statistics using a probability based on area fraction of grains. The result (Figure 4b) shows that, a nanograined layer with rather consistent modal boundary spacings ranging within 96 ± 11 nm has developed in the depth range of 0–30 μm. From a depth of 30 μm to 70 μm, the boundary spacing gradually increases to about 300 nm.

For a depth beyond 70 μm , the boundary spacing increases more steeply as the depth increases, with a boundary spacing of 1.9 μm observed at the depth of 90 μm . Note that Figure 4 only covers depths down to 110 μm from the surface. The microstructure at deeper depths is also deformed up to a depth of about 1 mm but is not covered in Figure 4.

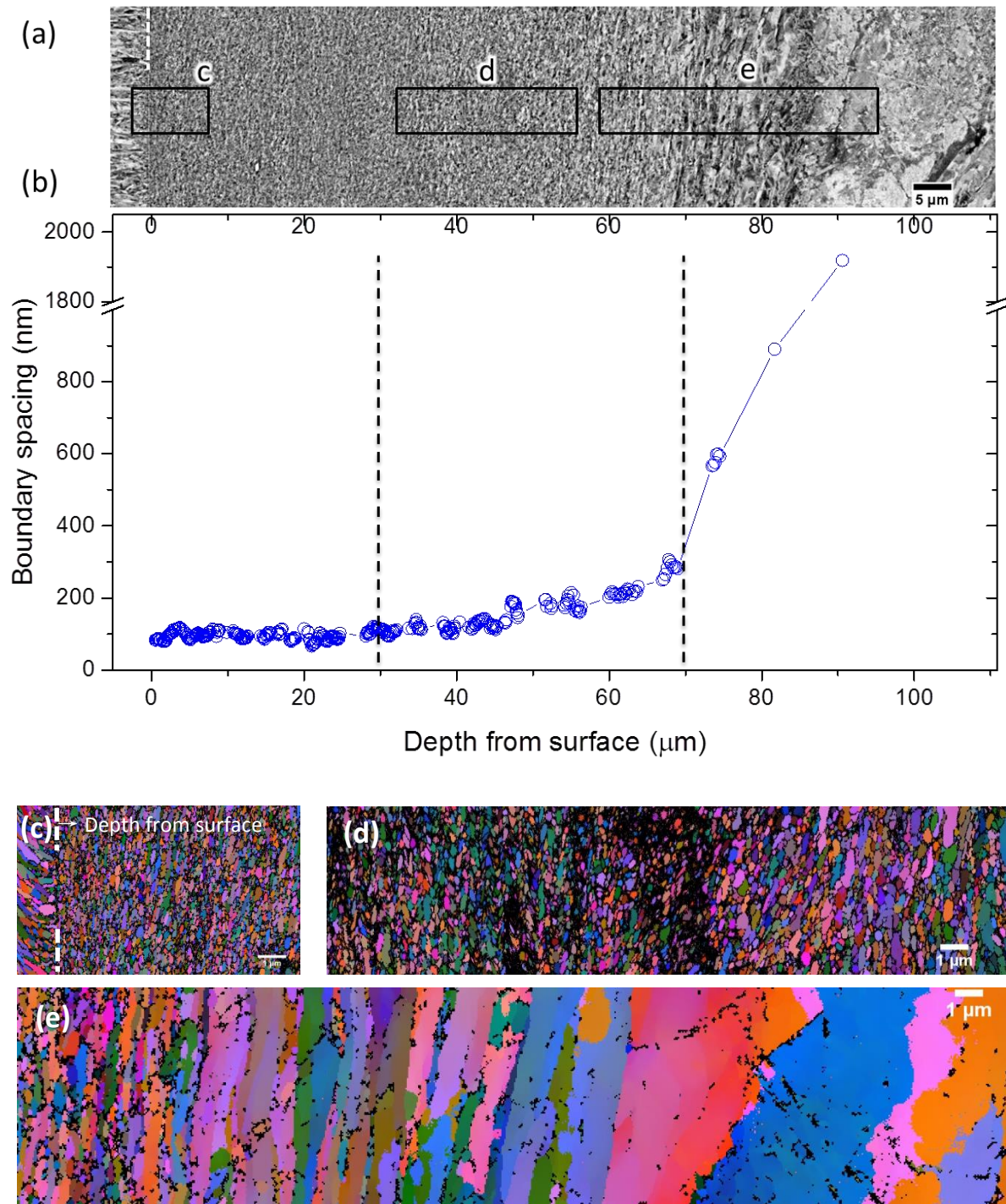


Figure 4. (a) An ECC image of the dark gray surface layer in the depth range of 0–110 μm from surface. The dashed line marks the surface of the plate. (b) Variation of the boundary spacings as a function of the depth from surface, as measured from EBSD maps. The two dashed lines separate regions of different gradients of the plot. Note that the image in (a) and the x-axis in (b) are on the

same scale. (c–e) Typical EBSD maps at various depths from regions marked by rectangles in (a). Note that (c–e) are on the same scale, and (c) is identical to Figure 3a.

The gradient nanostructured sample obtained in the present work may give opportunities to study the effects of microstructural parameters on the thermal behaviour. For example over a depth range of 1 mm the modal boundary spacing decreases from tens of micrometers to below 100 nm and the hardness increases to about 2.2 GPa which is 3.8 times the hardness of the fully recrystallized copper. As to the fraction of high angle boundaries (which are not shown in the present work) they also show a significant increase as the surface is approached. These microstructural changes reflect a significant increase in stored energy reducing the thermal stability in the surface and subsurface layers. This reduction may not be directly important for applications but it gives the opportunity to study kinetics of recovery and recrystallization in the finest scale and also as a function of the distance from the surface. To perform such a study microstructural information obtained by ECC/EBSD must be supplemented by transmission electron microscopy analysis, which can give information for example about triple junction motion [7] as the driving pressure will vary with the distance from the surface. Another study will be formation and growth of recrystallization nuclei into a microstructure with a gradient in driving pressure and differences in boundary characteristics. From an application point of view it may be possible by light annealing to soften the surface and increase its tribological properties while maintaining the advantages of a structure with a gradient from the subsurface to the center.

4. Conclusions

A technique called incremental frictional sliding has been developed and applied to process the flat surface of a Cu plate. The processed plate shows a hardened surface layer as thick as 1 mm with gradients both in hardness and in microstructure. In the topmost surface layer the hardness is increased to 2179 ± 31 MPa as compared with the initial hardness of 568 ± 10 MPa, and a nanostructure is developed. This gradient microstructure is difficult to quantify when it comes to the relation between microstructure, strength, and thermal behavior. However new and important information can be gained about the general behavior of metals on a structural scale from the micrometer to the nanometer dimension.

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